



TEAM stage: opportunities for in-situ microscopy

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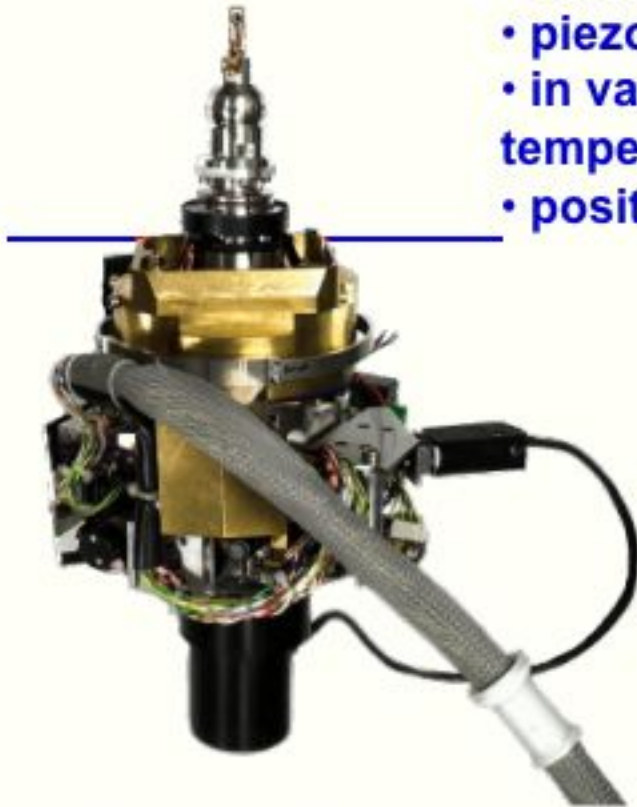
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Thomas Duden
Nord Andresen

NCEM,
Lawrence Berkeley National Lab



Advantages of the TEAM stage

- small size - highest stability
- piezo positioning technology
- in vacuum - pressure and temperature stability
- position sensing



Compustage

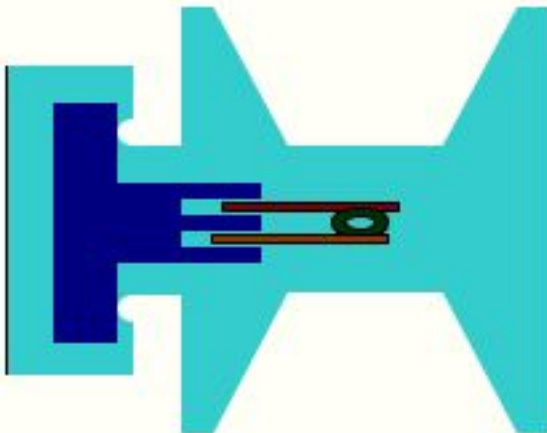


TEAM stage



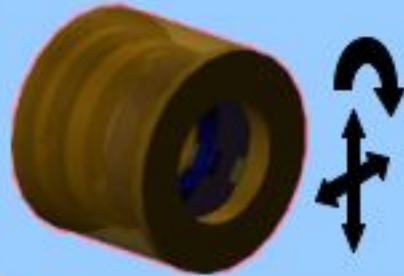
Modular design with four moving parts

5-axis piezo stage



four moving parts

alpha module



Three axis "alpha module" provides y -, z -motion, α -tilt

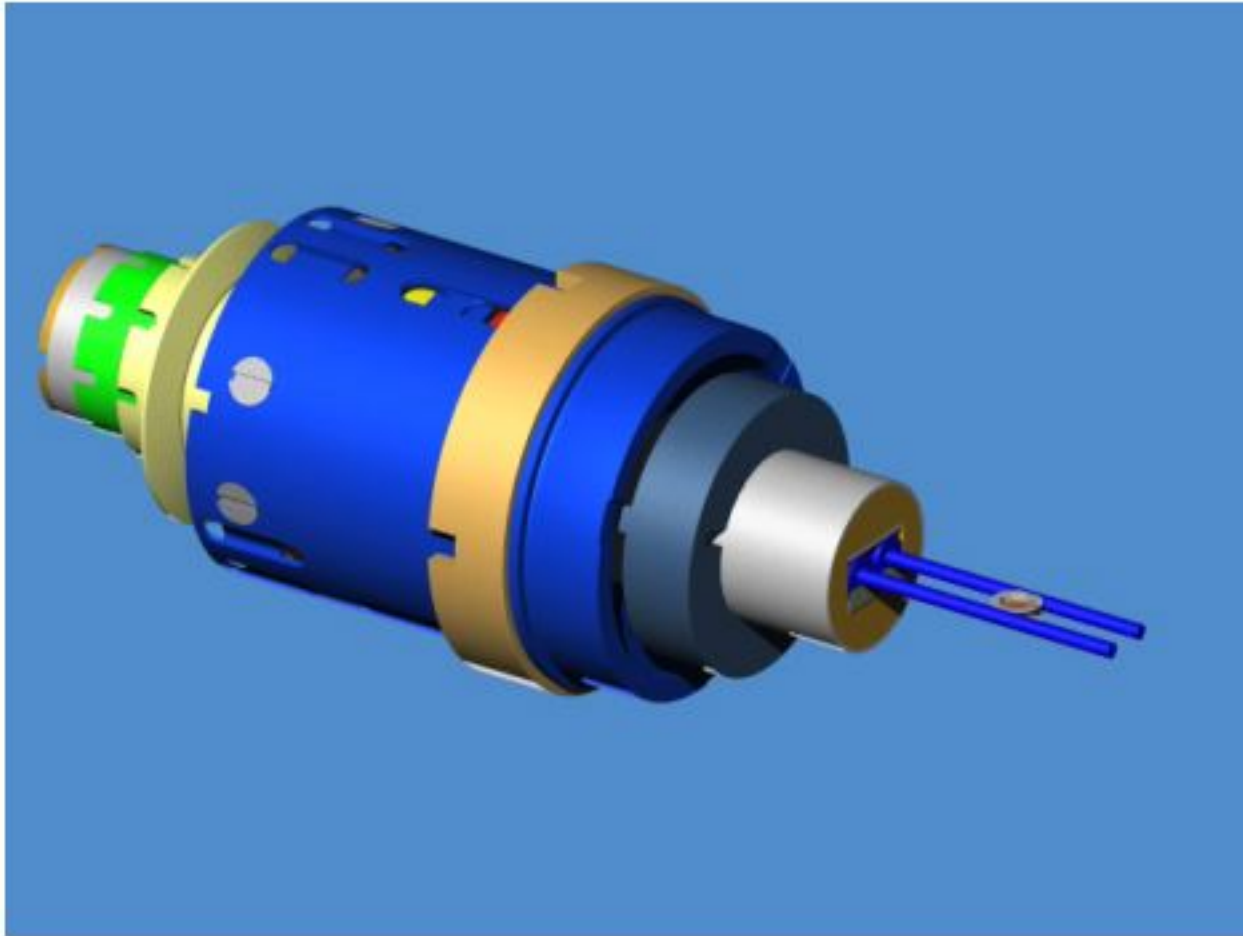
beta module



Two axis, interchangeable "beta module" provides x -motion, γ -tilt (rotation)



the alpha stage in action





Modular design maximizes flexibility

Beta module designs



Compustage
~0.3 kHz

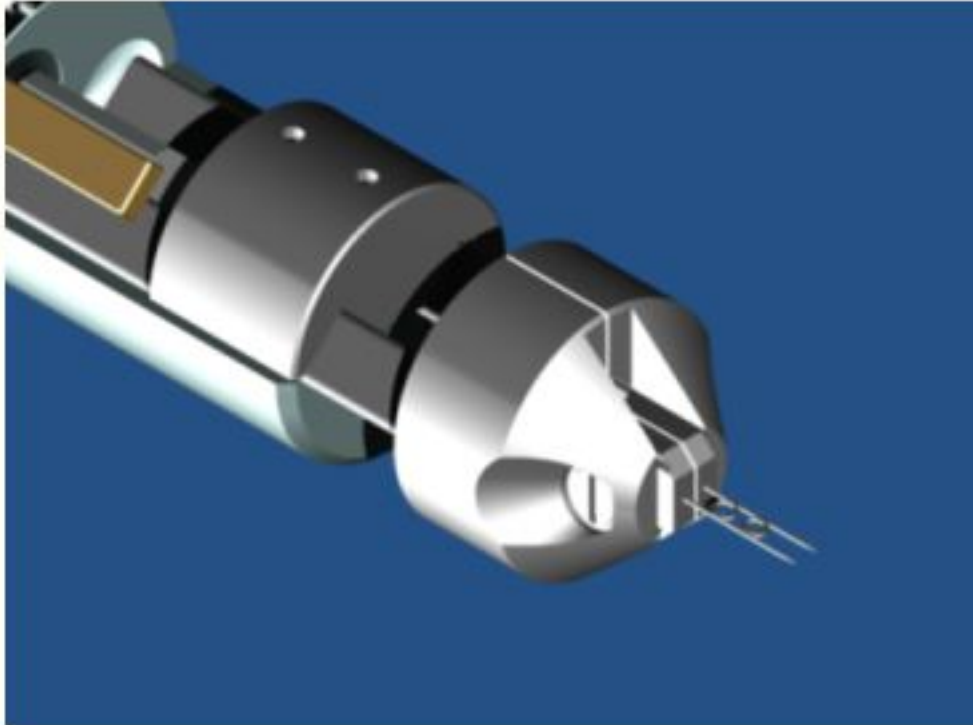
Attoboy
1.8 kHz
1.2 mm grid

Lily
1.2 kHz
2.3 mm grid

Rhonda
1.2 kHz
3 mm grid



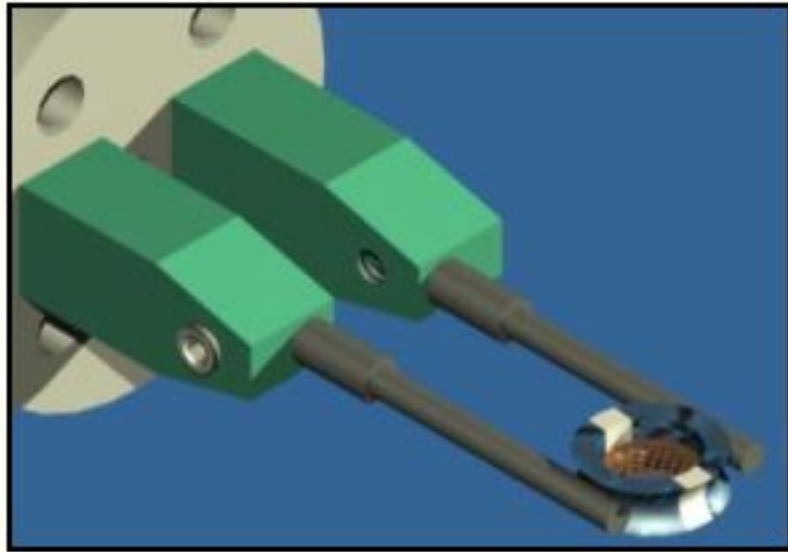
Attoboy beta module



- **highest stability**
- **high tilt angles:**
tomography in TEAM
0.5 within 2 mm
- Small sample size (<1 mm)
experimentation difficult



Biasing stage requirements



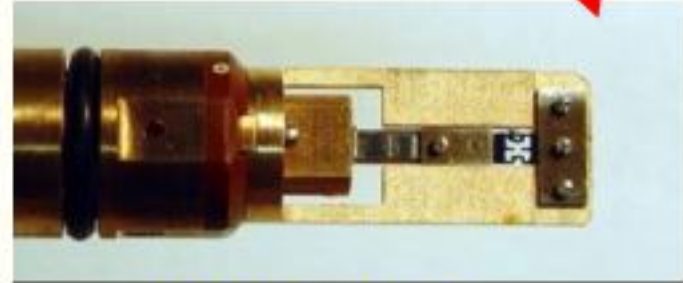
- retain high stability, piezoelectric stack motors
- in-situ, capacitive encoders
- larger sample size -- 3 mm grids
- prioritize in-situ biasing for experimentation



In-situ modules - single-tilt



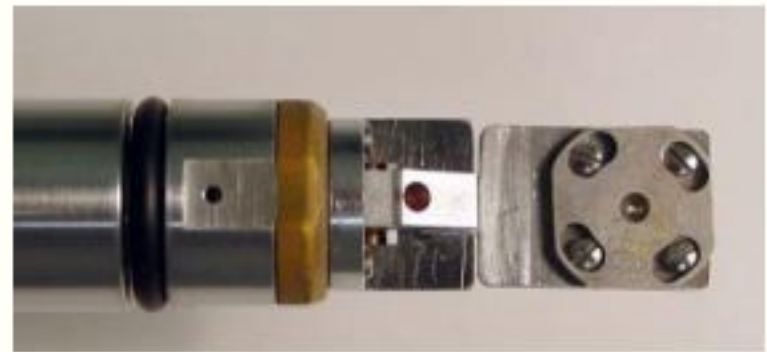
Standardized 8-pin MEMS stage



MEMS tensile stage



Nanomanipulator



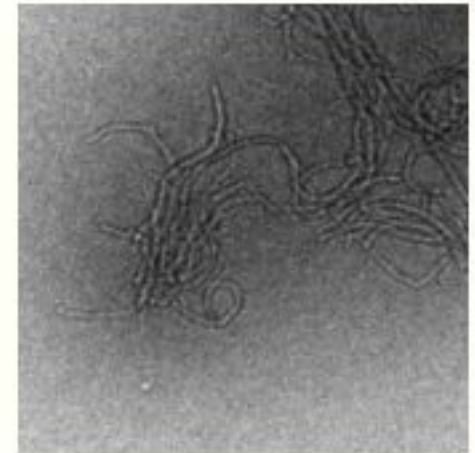
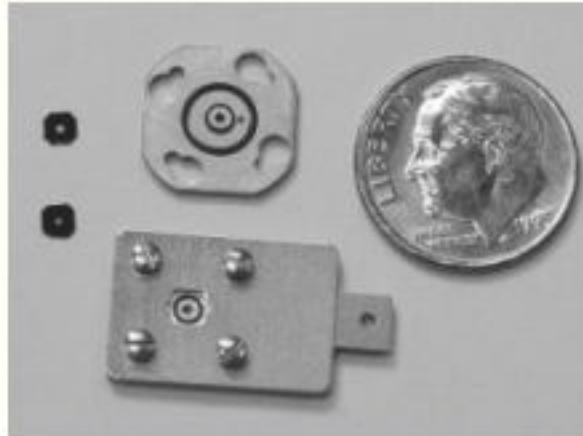
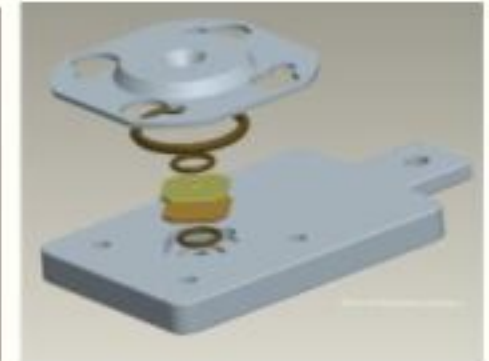
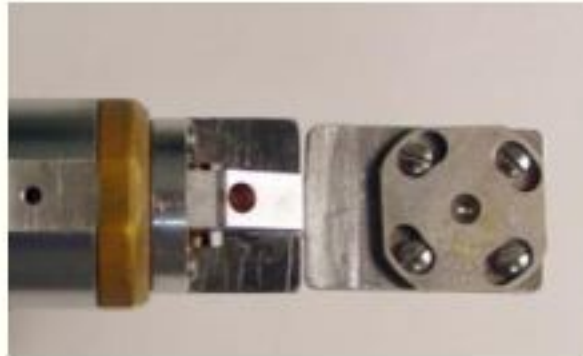
Liquid Cell

TEAM R@D



Liquid cell

- Liquid is sealed between two SiN_x membranes.
- O-rings seals the grids. No glue needed.
- Rotating cap for quick assembly.
 - takes less than 5 min to assemble!
- Gap between two membranes can be controlled down to 200nm.



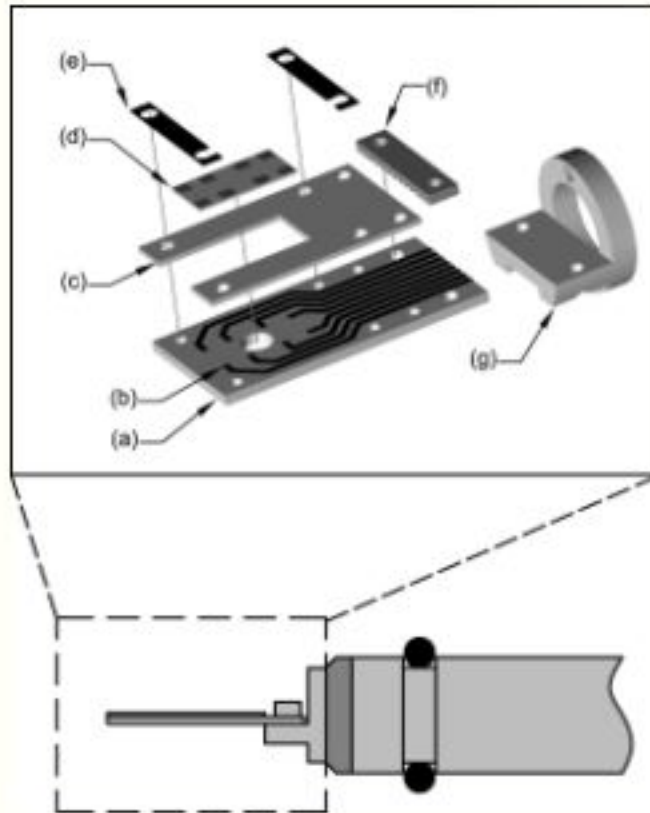
carbon canotubes in water

Wen, Marsh, 2008

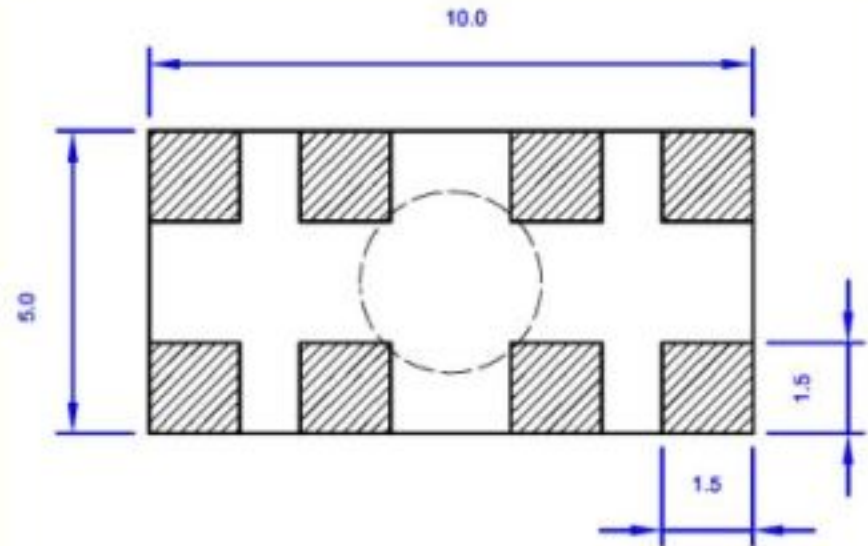


8-pin MEMS holder

Holder



MEMS device outline

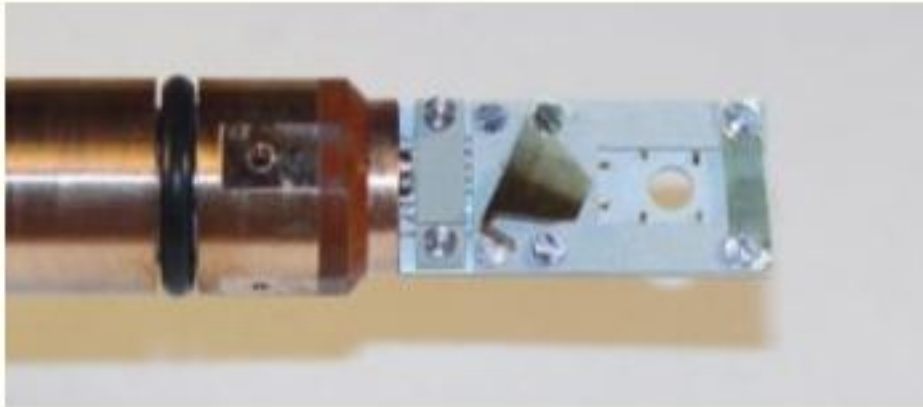


The goal was to create a standardized platform for which anyone can design a device.

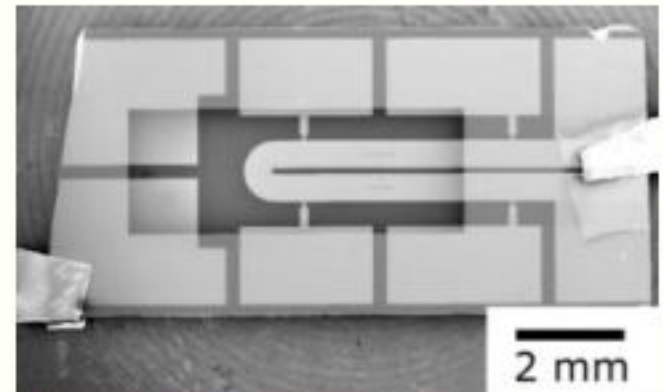
USPTO Application 20060025002 (2005).



Standardized MEMS Various users

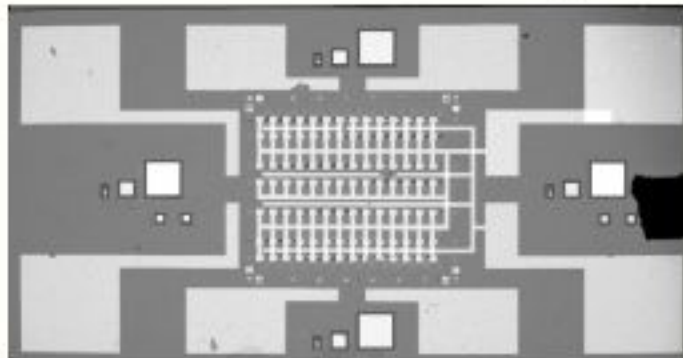


Nanocalorimeter



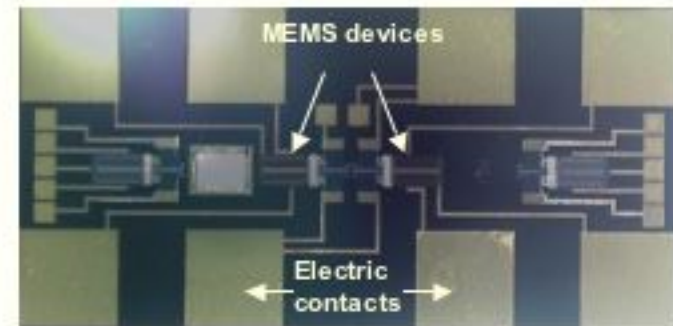
J Mater. Res. **20**, 1802 (2005).

Transistor biasing



Kim, Olson, *et al.* *APL*. **87**
173108 (2005).

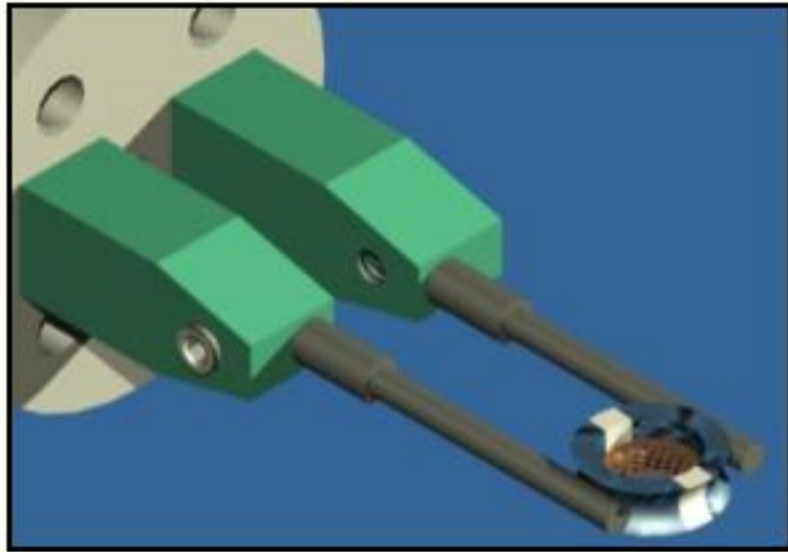
Tensile testing



Yong, Moldovan, and Espinosa.
APL. **86** 013506 (2005).



Biasing stage requirements



- retain high stability, piezoelectric stack motors
- in-situ, capacitive encoders
- larger sample size -- 3 mm grids
- prioritize in-situ biasing for experimentation



Prototype/testing biasing stage

Electronics incorporated into the holder



LED off



LED on



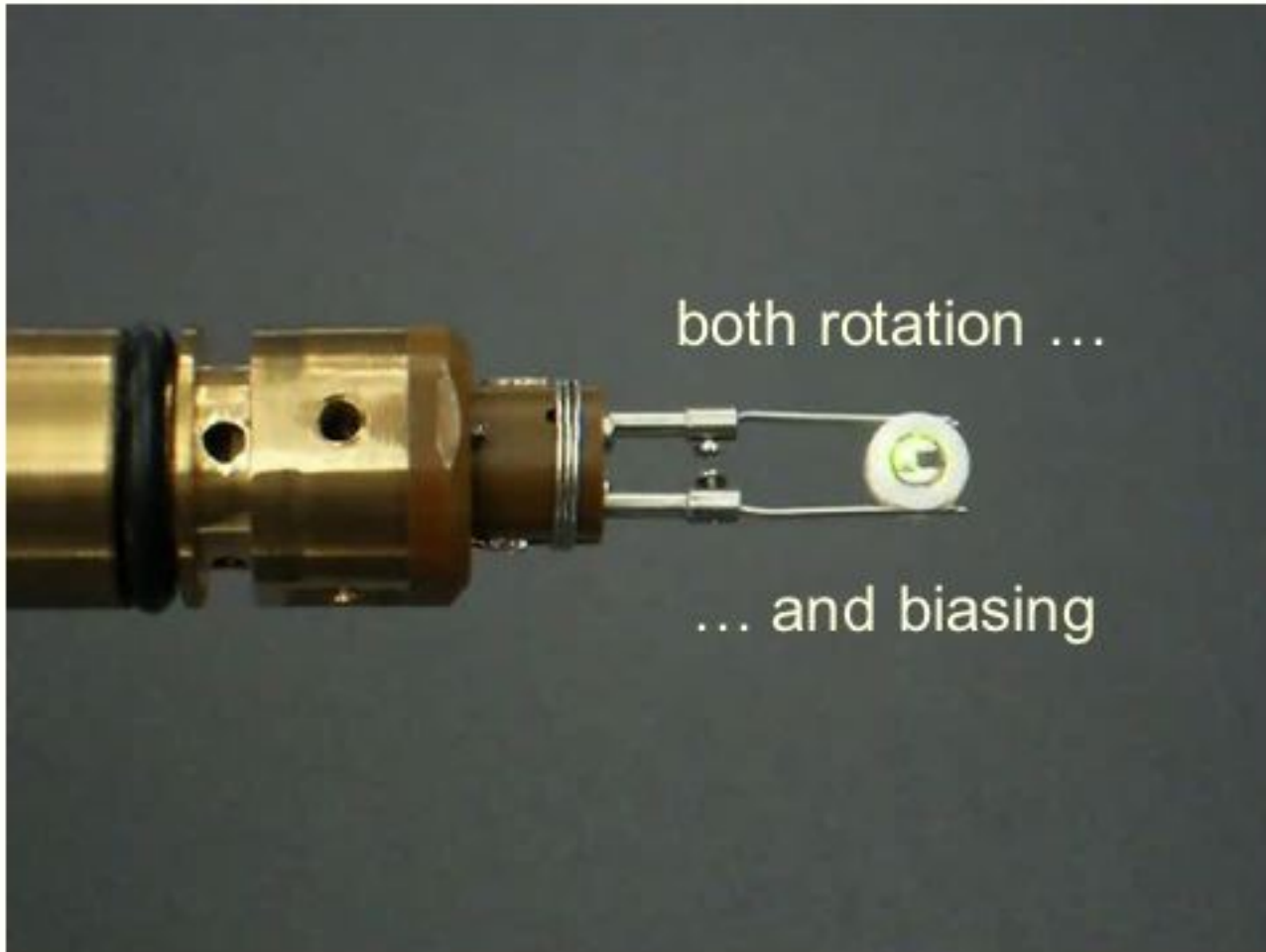
I2C-to-serial
interface board,
inside the handle

analog-to-digital
board,
in vacuum!

x, γ motion
plus
electrical contact



Biasing stage

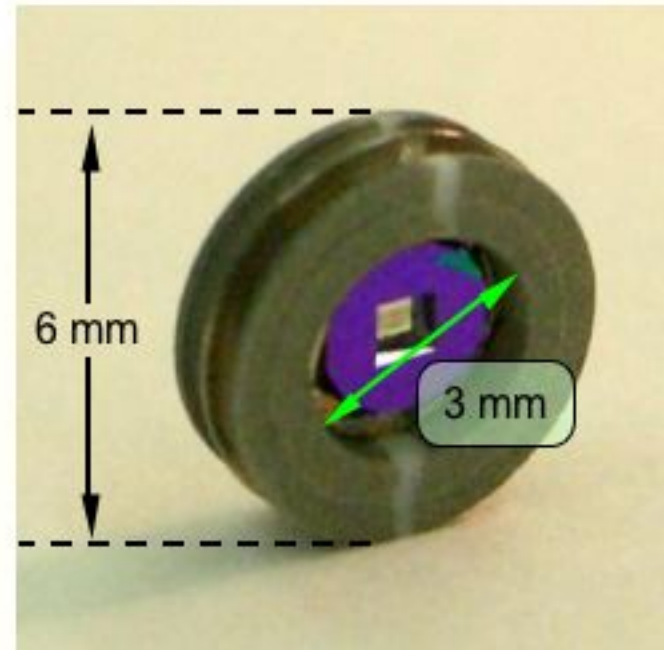
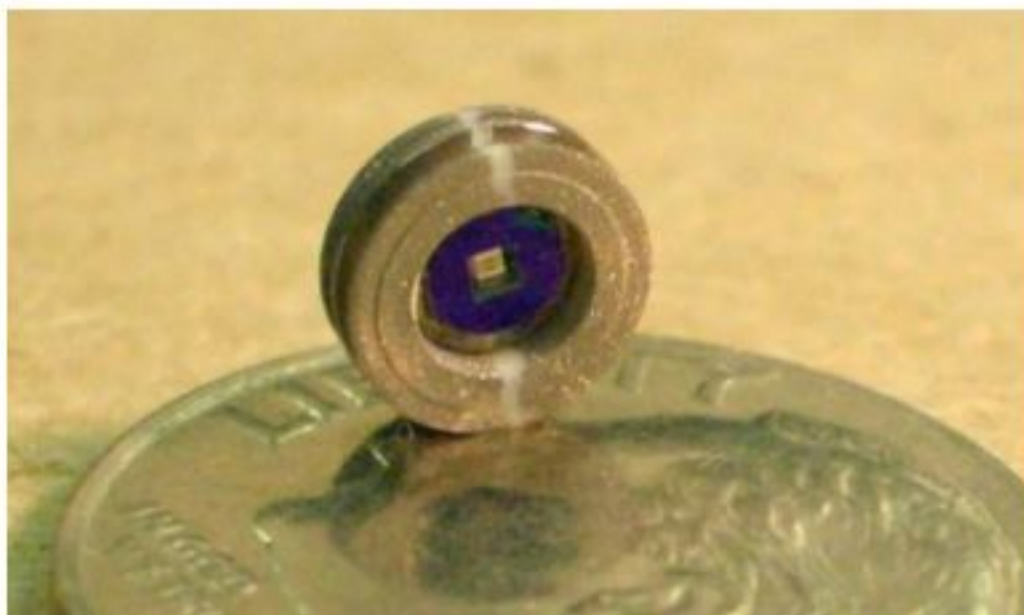




Sample holder

Should be:

- insulating (prevents shorting of device)
- conductive (prevents charging in microscope)
- able to take a standard 3 mm grid





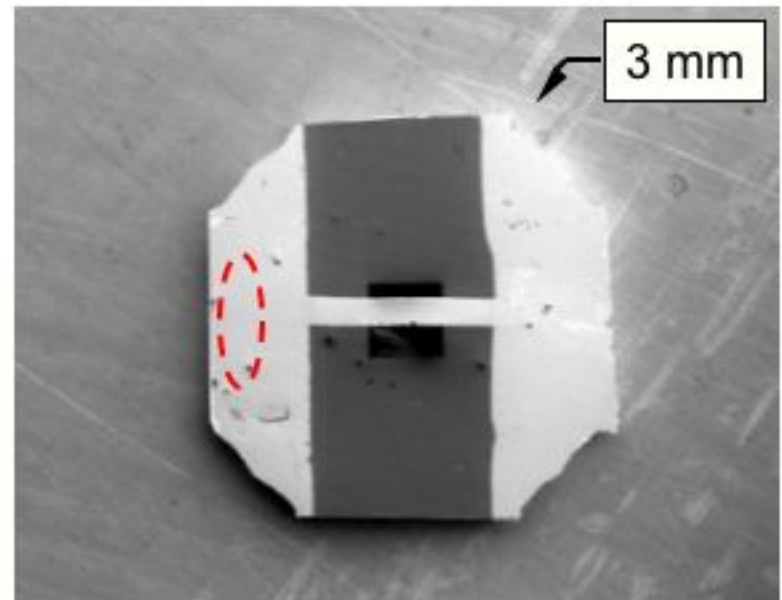
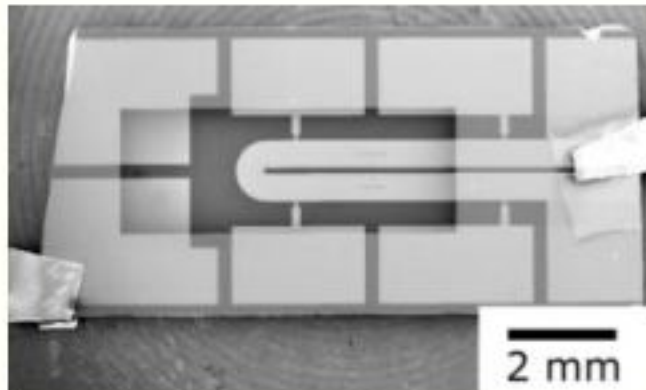
In-situ heating, two devices:

- Designed at FS-MRL
 - Based around off-the-shelf Si_3N_4 grids
 - Patterned metal heater strip
- Protochips design
 - Ceramic membrane
 - Coming soon to a microscopy supplier near you



Microfabricated heater #1

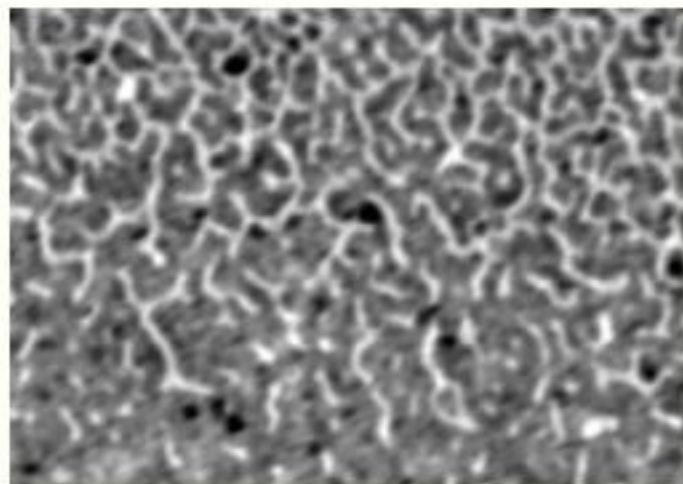
- Made using commercial Si_3N_4 grids
- 30-50 nm membrane thickness
- Maximum temperature: $\sim 350^\circ\text{C}$
- Standard 3 mm form factor



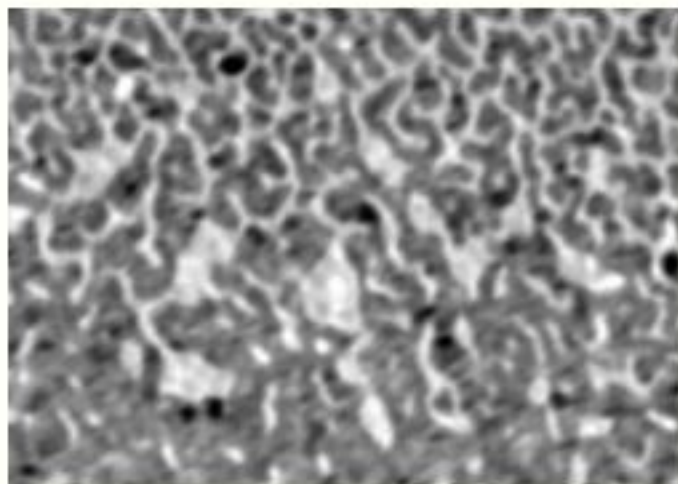


Heating

Heating causes rearrangement of a Bi metal film



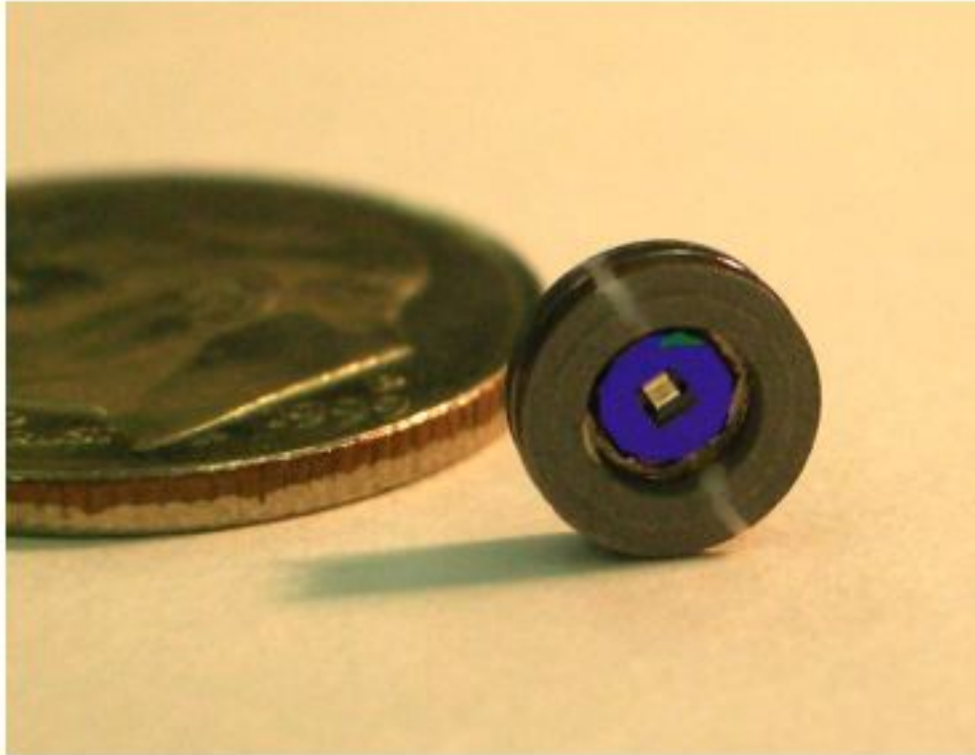
room temp.



~300°C



Microfabricated heater #2



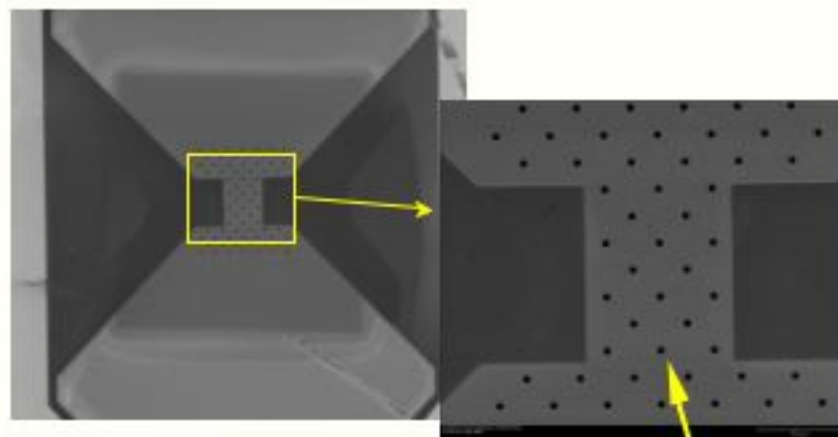
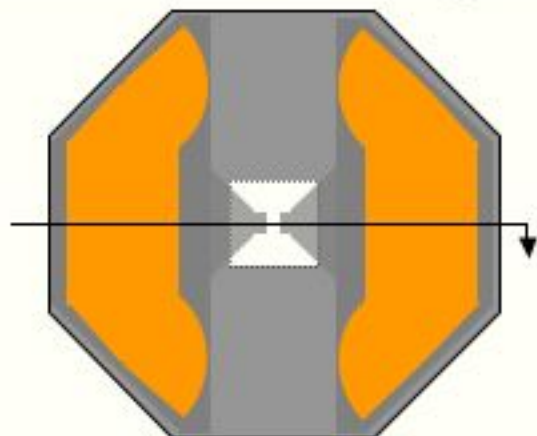
- Made by Protochips
- Maximum temperature: $\sim 1200^{\circ}\text{C}$
- ~ 3 mm in size
- requires a few mA to operate



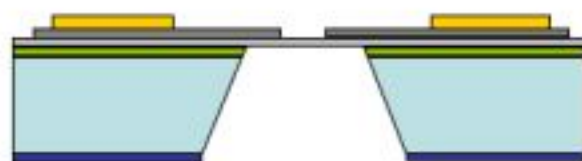


Protochips AduroTM Technology

Aduro technology places MEMS-based microheaters directly within the TEM. Current forced through a thin membrane provides rapid Joule heating.



an array of holes
in the membrane
for clear viewing



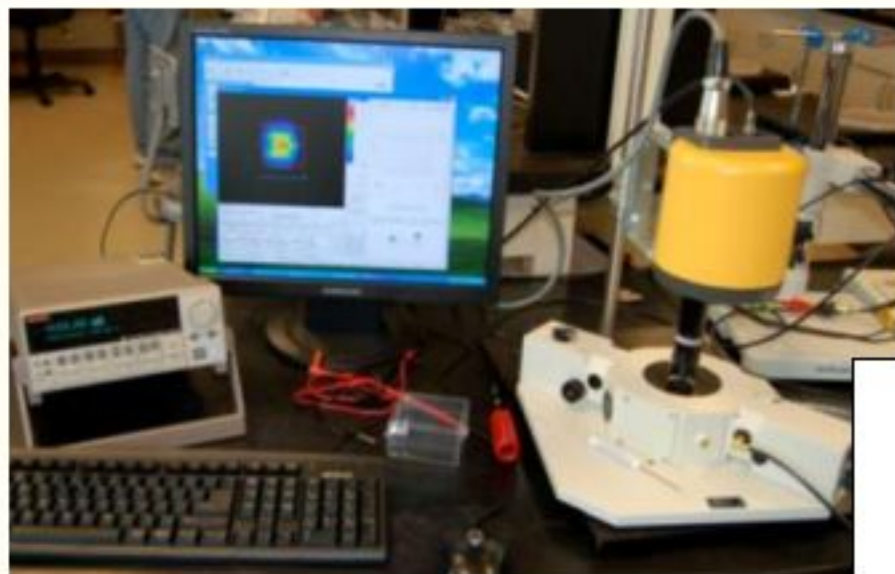
- Ti/Au contact
- High-conductivity ceramic
- Low-conductivity ceramic
- Silicon dioxide
- Silicon
- Silicon nitride



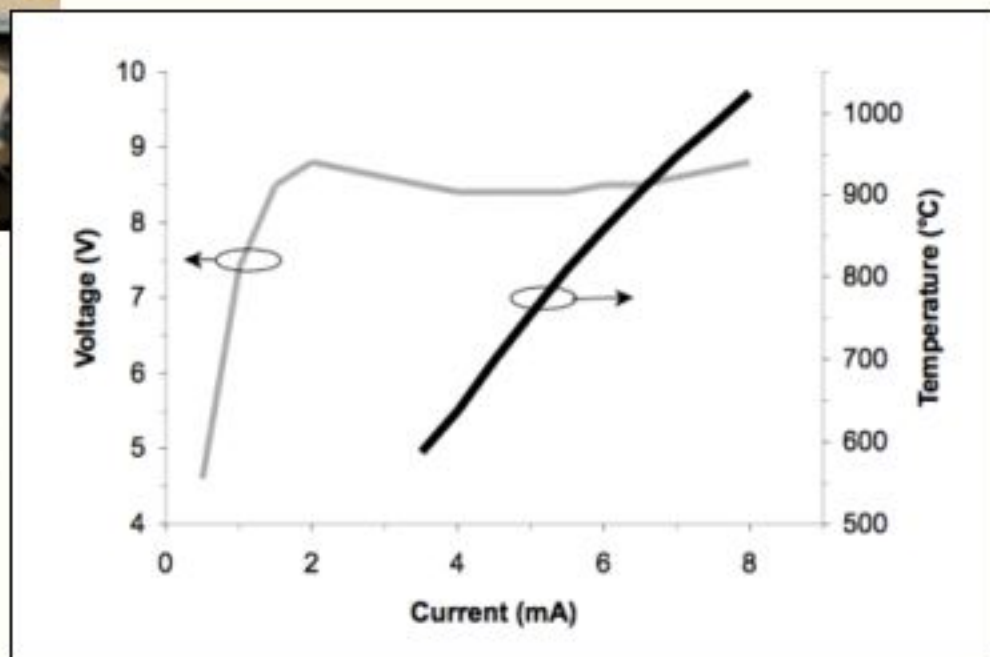
ProtochipsTM
interactive real-time microscopyTM



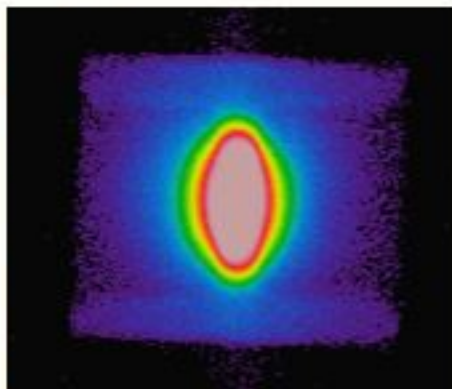
Heater calibration



Each microheating device is characterized under vacuum using a Mikron M9104 Ultra High Resolution Imaging Pyrometer with microscope optics



optical



thermal

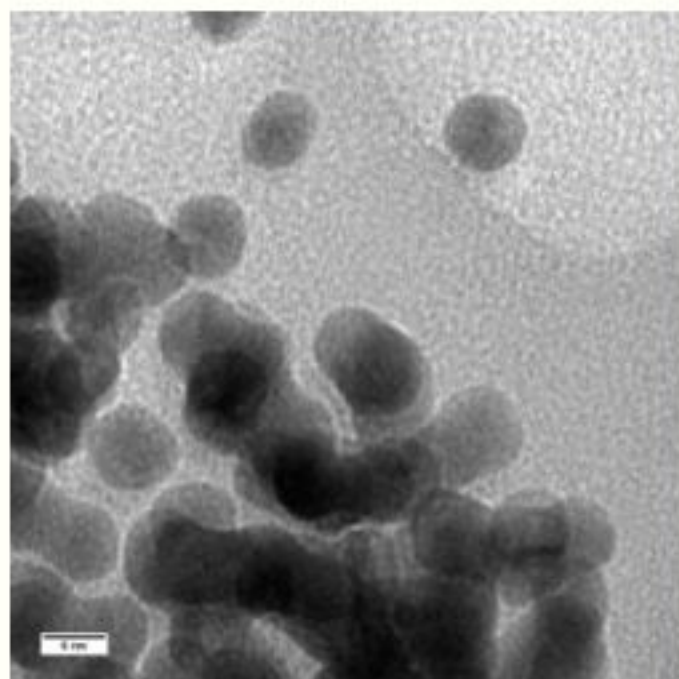


Protochips

interactive real-time microscopy™



In-situ heating

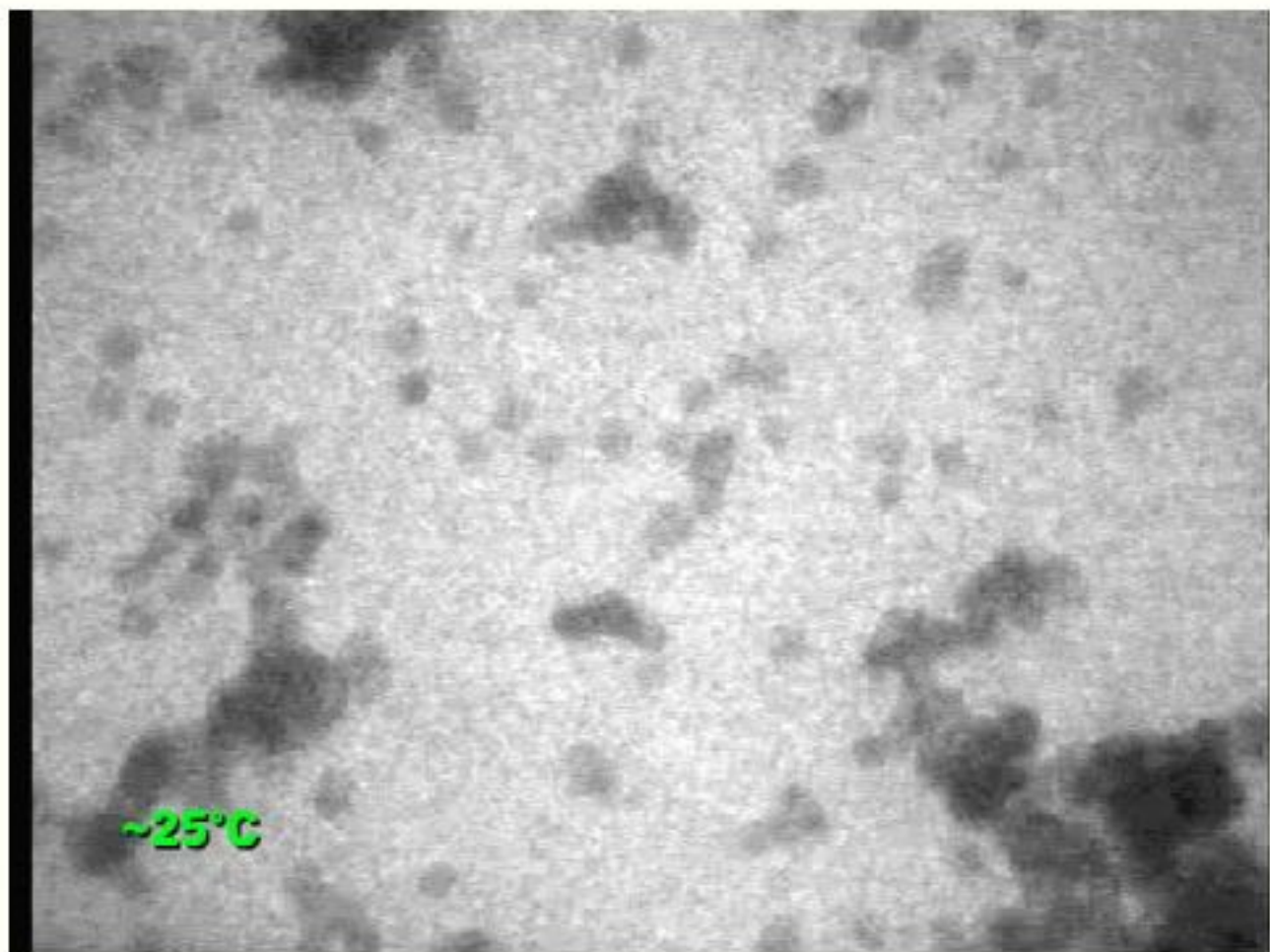


- Start with colloidal Au nanoparticles ~ 10 nm in size
- Particles on a carbon film on a Protochips device
- Heat and observe



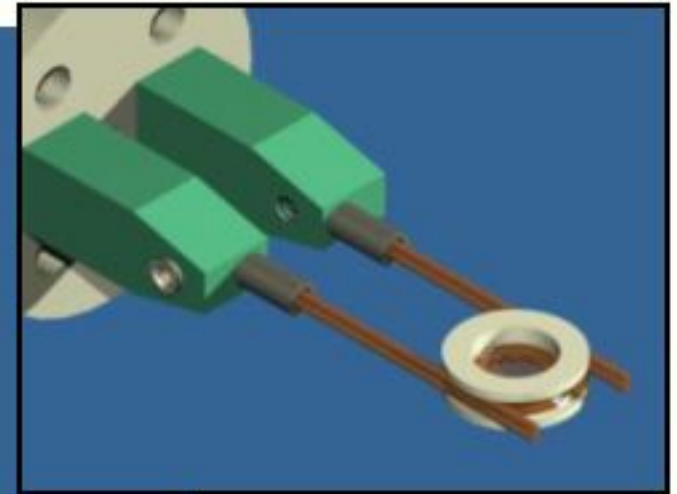
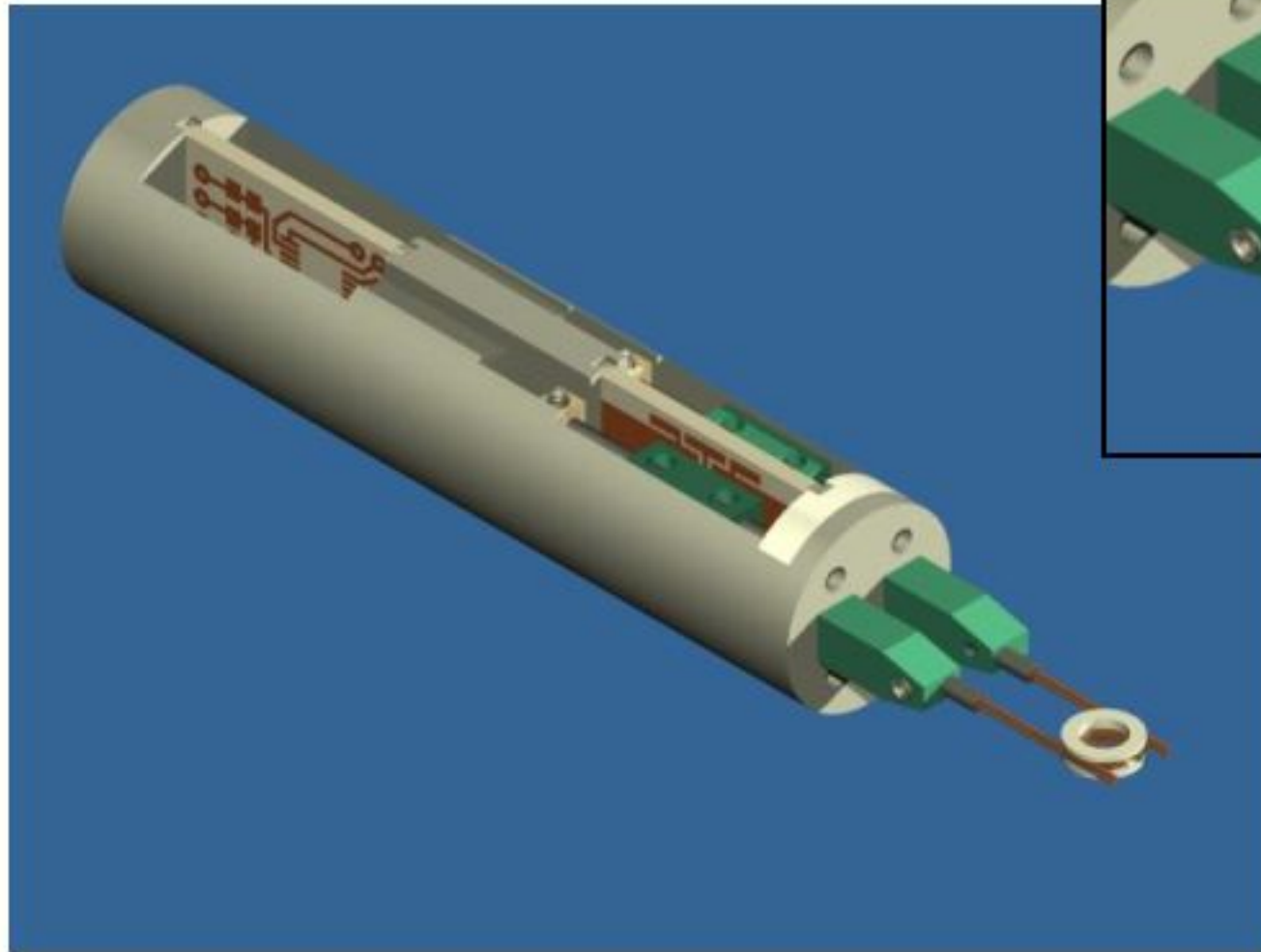


In-situ heating, Au melting





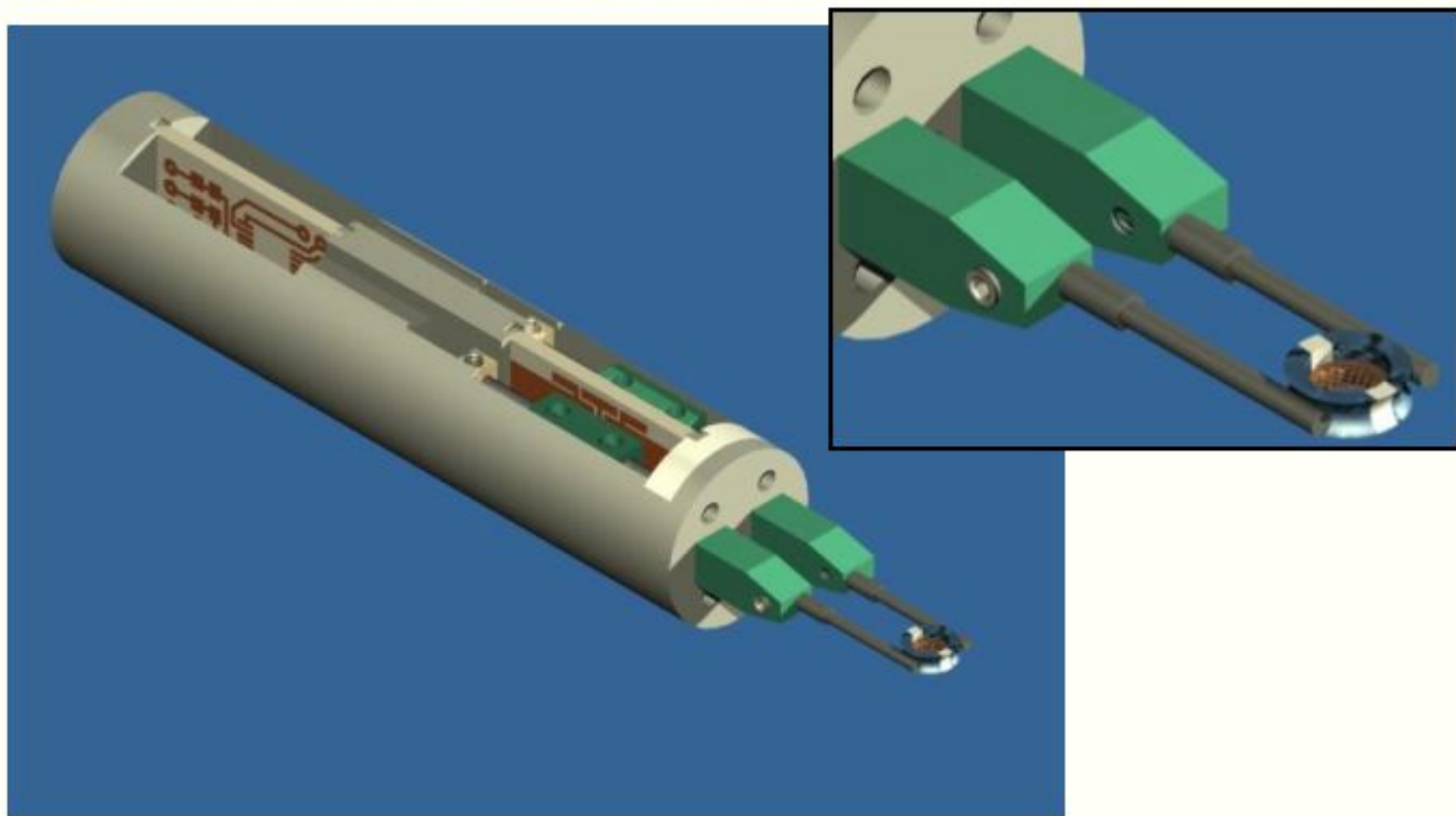
In-situ TEAM stage



- 2-contact sample cartridge
- interchangeable rods
- capacitive encoders
- in-vacuum electronics

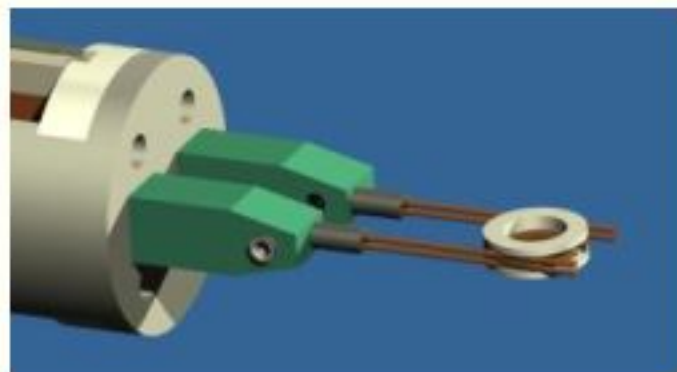


In-situ TEAM stage





Optimization of the rods/cartridge assembly



$$f_{\text{est.}} \cong 1000 \text{ Hz}$$



$$f_{\text{est.}} > 1500 \text{ Hz}$$

$$f_0 = \frac{1}{2\pi} \sqrt{\frac{3EI}{(0.22M_{\text{beam}} + M_{\text{weight}})L^3}}$$



different designs may affect

- automatic loading
- resonance frequency



TEAM stage: opportunities for in-situ microscopy

1

finest motion
no in-situ



Attoboy TEAM stage

2

fine motion
in-situ biasing/temp.

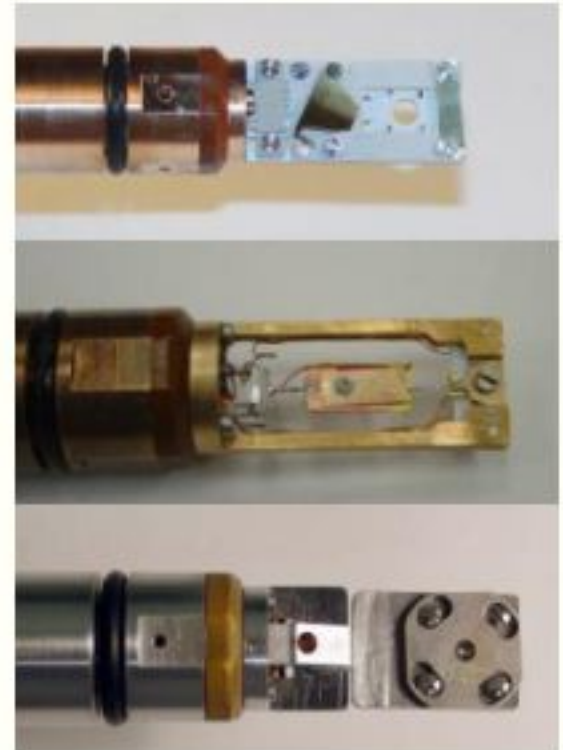


Biasing TEAM stage



3

single tilt
lots of in-situ capabilities



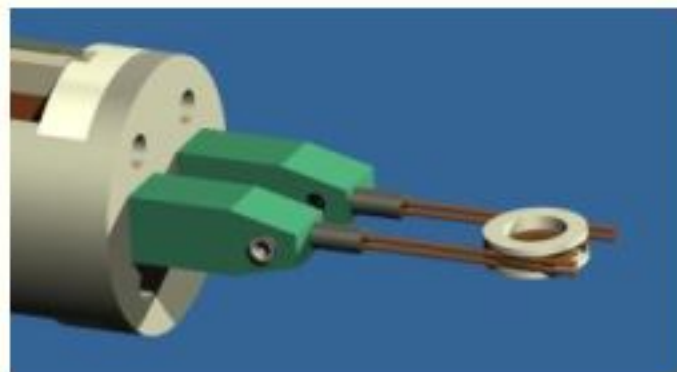
platform for user to develop their experiments
proof of principle in conventional TEMs



Thank you



Optimization of the rods/cartridge assembly



$f_{\text{est.}} \cong 1000 \text{ Hz}$



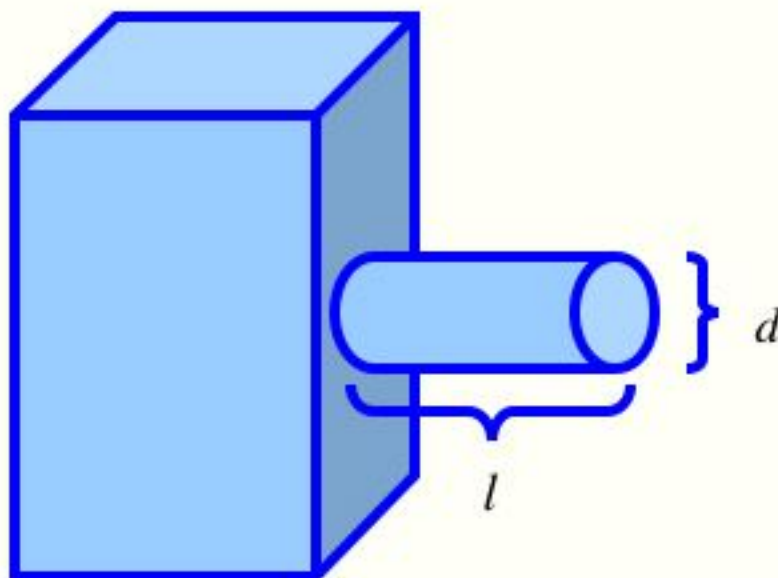
$f_{\text{est.}} > 1500 \text{ Hz}$

different designs may affect

- automatic loading
- resonance frequency



Ballpark-estimate of best possible resonance



$$\omega_{Stage} \approx \frac{1}{10} \frac{d}{l^2} v_{sound} \approx 0.1 \times \frac{0.01m}{0.05m^2} \times 5500 \frac{m}{sec} \approx 2000Hz$$

- a) Reaching ~ 2000 Hz should be feasible within reasonable geometric constraints of several centimeter linear dimensions
- b) Aiming above 2000 Hz would be unreasonable -- ~ 2000 Hz is also the resonance of a typical TEM objective, i.e. relative motion of upper- vs lower polepiece would simply dominate a hypothetical, even stiffer stage



In-situ microscopy

lab in the microscope

Long-term TEAM goal is to enable in-situ experiments.

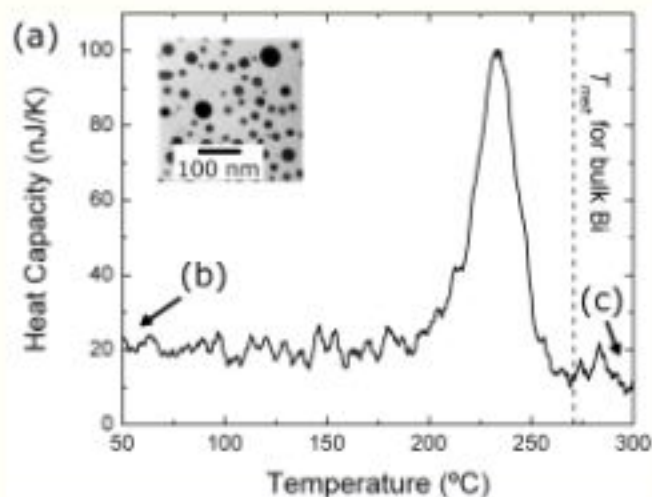
Currently have five holders for use in CMM microscopes:

1. MEMS
2. Wet-cell
3. Tensile holder
4. Nanomanipulator
5. Biasing



In-situ heating and melting

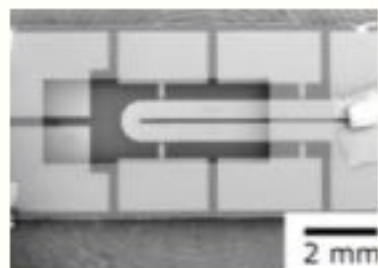
Nanocalorimetry



Bi nanoparticles
heating rate 30,000 K/sec

Zhang, Olson, *et al.*,
J Mater. Res. **20**, 1802 (2005).

Sensor



Slow heating

